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Auteurs: Xavier Lachapelle-Trouillard, Michel Labrecque, & Yves Comeau
Authors:

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Title: Treatment and valorization of a primary municipal wastewater by a short rotation willow coppice vegetation filter

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Authors and affiliations:

Xavier Lachapelle-T.^{a,1,*}, Michel Labrecque^b, Yves Comeau^a

^aDepartment of Civil, Geological and Mining Engineering
Polytechnique Montréal
2900 Edouard-Montpetit Boulevard
Montréal, Québec, Canada
H3T 1J4
xavierlt@agroenergie.ca
yves.comeau@polymtl.ca

^bInstitut de recherche en biologie végétale (IRBV)
4101 Sherbrooke East
Montréal, Québec, Canada
H1X 2B2
michel.labrecque@umontreal.ca

*Corresponding author

¹Present address : Agro Énergie, 517 Rang du Ruisseau des Anges Sud, Saint-Roch-de-l'Achigan, Québec, Canada, J0K 3H0, xavierlt@agroenergie.ca

ABSTRACT

The objective of this study was to evaluate the treatment efficiency of a short rotation willow coppice (SRWC) vegetation filter for the treatment of wastewater from a municipal primary effluent in a humid continental climate context. The experimental work was carried out at pilot scale on a willow plantation located in Québec, Canada. The experimental design included nine plots that were irrigated with groundwater ($L0 = 14$ mm/d) or two primary effluents ($L1 = 10$ and $L2 = 16$ mm/d) for 111 days. This research showed that SRWCs operated on coarse-textured soils allow efficient removal of organic matter (91 % of COD for $L1$ and $L2$) and nitrogen (98 % of TKN for $L1$ and $L2$) from wastewater. It was also shown, in this case, that the total nitrogen loading should be used as the limiting design parameter to minimize the risk of contaminating underground drinking water sources with nitrates. Almost complete removal of total phosphorus was observed during this experiment (98 % for $L1$ and $L2$). However, a significant increase in soil available phosphorus was observed following the $L2$ treatment, which suggests an eventual phosphorus soil profile saturation in the event of continued wastewater irrigation. Avoiding such a saturation would require chemical phosphorus removal upstream of SRWC vegetation filters. Finally, an imbalance between irrigation and willows needs was observed as a result of irrigating plots at a constant hydraulic loading rate. Thus, irrigation of an SRWC with wastewater should be modulated according to willow seasonal transpiration trends to allow a better allocation of water and nutrients according to plant needs, and in doing so, increase treatment efficiency and resources valorization.

Highlights

- Efficient removal of OM and nitrogen (TKN) by coarse textured SRWC vegetation filter
- TN should be used as the limiting design parameter of SRWC vegetation filter
- Eventual P soil profile saturation in the event of continued wastewater irrigation
- Irrigation should be modulated according to willow seasonal transpiration trends

KEYWORDS

Willow; short rotation coppice; vegetation filter; municipal wastewater; wastewater treatment; resource recovery

1. Introduction

Many small and isolated North American communities fail to effectively treat their wastewater due to lack of funds, skilled personnel or maintenance experts (Joy et al., 2003). In response to this issue there is a need to continue to develop extensive wastewater treatment systems that require low construction and operating costs. The use of short rotation willow coppice (SRWC) vegetation filter to treat wastewater constitutes a potential solution to this issue and an innovative way of water resource recovery.

In addition to their high rates of evapotranspiration, willows have several features that make them suitable for a variety of environmental applications that can be achieved in addition to biomass production. These characteristics include their non-edibility, their high nitrogen absorption capacity as well as their ability to absorb certain metals such as cadmium (Aronsson and Perttu, 2001). Willows have been used for a multitude of environmental applications, such as municipal effluent treatment (Dimitriou and Aronsson, 2011; Guidi Nissim et al., 2015; Hasselgren, 1998; Holm and Heinsoo, 2013; Larsson et al., 2003; Perttu and Kowalik, 1997), biosolids treatment (Dimitriou and Aronsson, 2004), landfill leachate treatment (Aronsson et al., 2010; Dimitriou and Aronsson, 2010), polluted groundwater (Guidi Nissim et al., 2014) and effluent total evapotranspiration (Curneen and Gill, 2014, 2016; Frédette et al., 2019; Gregersen and Brix, 2001) for more than twenty years. In recent years, it has also been shown that the use of willow beds has an interesting potential for the treatment of wastewater (Grebenschykova et al., 2017, Khurelbaatar et al., 2017), complete evapotranspiration of effluent (Frédette et al., 2019) and nutrient recovery (Rastas Amofah et al., 2012) in cold climate. The similarity between willow N, P and K proportional

requirements (100:14:72) and the proportion of these nutrients typically found in municipal wastewater (100:18:65) makes the use of SRWC particularly appealing for the treatment of this type of effluent (Perttu, 1993).

Most studies treating of the use of SRWC for the treatment of municipal wastewater have been carried out in Europe using European willow cultivars, such as *Salix viminalis* (Hasselgren, 1998; Holm and Heinsoo, 2013), 'Jorr' (Larsson et al., 2003) or 'Tora' (Dimitriou and Aronsson, 2011; Holm and Heinsoo, 2013). The effectiveness of SRWC vegetation filter for the treatment wastewater from a municipal primary effluent under a North American climate and using willow cultivars adapted to American and Canadian pedoclimatic conditions (*Salix miyabeana* 'SX64' and 'SX67', *Salix purpurea* 'Fish Creek', etc.; Smart et al., 2010) has not yet been evaluated. Moreover, the removal efficiency of organic matter (COD or BOD₅) and ammonia, two important constituents of wastewater, are generally poorly addressed in the literature, which rather deals with the removal of total nitrogen and total phosphorus.

The objective of this study was to evaluate the treatment efficiency of an SRWC for the treatment of wastewater from a municipal primary effluent in a humid continental climate context. It was desired to develop design and operation criteria for this process from the perspective of a North American application.

2. Material and methods

2.1 Site description

The experimental work was carried out at pilot scale at the municipality of Saint-Roch-de-l'Achigan, Québec, Canada (45°51'29"N, 73°35'36"W, 52 m above sea level). The experiment took place on a two-hectare willow plantation located near the city water resource recovery facility (WRRF). *Salix miyabeana* 'SX67' was planted at a density of 16 000 plants/ha (1.83 m row spacing, 0.34 m plants spacing on row) in the spring 2008. The site was used for maize cultivation prior to the willow plantation establishment. A secondary effluent irrigation experiment was carried out at the plantation from 2008 to 2012 (Guidi Nissim et al., 2015, Jerbi et al., 2014). The plantation was last coppiced in the fall 2015. Soil was characterized at two depths (0-30 and 30-70 cm) before the start of the experiment (Table 1). For each of these two depths, composite samples were randomly collected at the experimental site. The soil was a loamy sand from 0 cm to 30 cm, a sand from 30 cm to 70 cm and a clay downward. The top layer was characterized as having a low total available soil water (8%; estimated according to Saxton and Rawls (2006)) and a high saturated hydraulic conductivity (1.4 E-02 to 2.0 E-03 cm/s; estimated according to Chapuis (2008)). Higher contents of organic matter, total organic carbon (TOC) and nutrients (except for K) were measured in the soil top layer than in the lower layer (analytical methods presented in section 2.4).

2.2 Meteorological conditions

The region has a humid continental climate with a high temperature amplitude. For the years 2006 to 2015, average daily minimal and maximal temperature at the nearest weather station

(L'Assomption, Québec; 45°48'34"N, 73°26'05"W; 14 km from site) were 1 ± 12 °C and 11 ± 13 °C and the yearly average precipitation was 1 090 mm, 55% of which occurred during growing season (180 days from May to October; Environment Canada, 2017). Average temperatures measured during the willow growing season (May 1st to October 31st; 16.5 °C) and the experimental irrigation period (July 20th to November 8th; 15.3°C) were slightly higher than the 2006-2015 normal (15.9 °C and 14.2 °C, respectively; Figure 1). Total rainfall during the growing season (565 mm) was lower than normal (606 mm), whereas rainfall during the irrigation period (390 mm) was higher than normal (350 mm). High rainfall events occurred during the irrigation season in mid-August and mid-October. Reference evapotranspiration (ET_o) values, as estimated with the FAO Penman-Monteith method (Allen et al., 1998), were slightly lower than total rainfall during the growing season (550 mm) and irrigation period (370 mm).

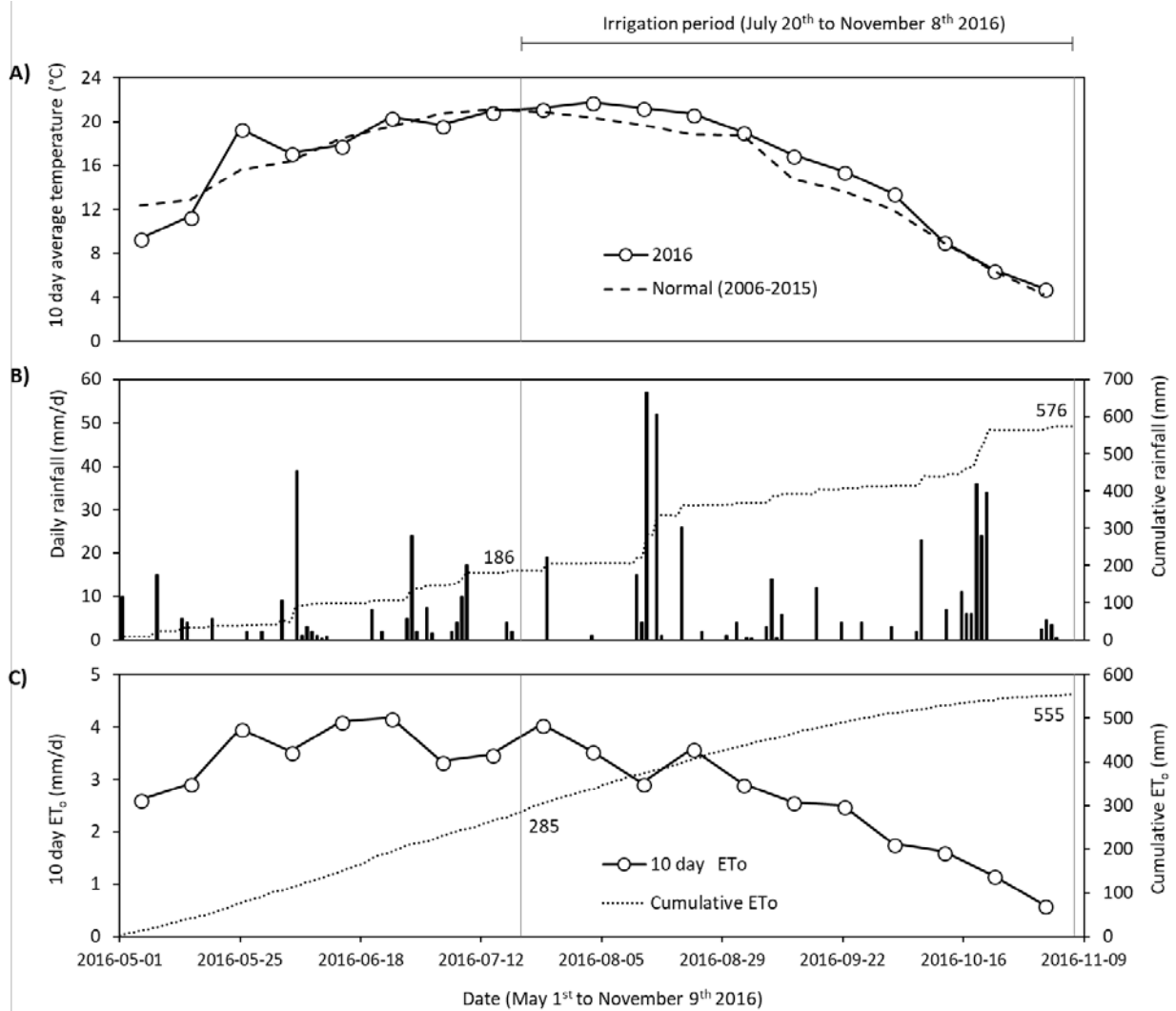


Figure 1. A) 2016 10 days average temperatures compare to 2006-2015 normal temperatures; B) Daily and cumulative rainfall; C) 10 days and cumulative reference evapotranspiration (ET₀) during the 2016 willow growing season.

Parameters	Units	Soil depth (cm)		n
		0-30	30-70	
Sand	wt%	79	88	3
Silt	wt%	17	8	3
Clay	wt%	4	4	3
Texture		Loamy sand	Sand	
Coefficient of uniformity		13.3	3.3	3
Coefficient of curvature		2.9	1	3
Bulk density	g/cm ³	1.4	1.38	3
Porosity	v%	48	50	3
Wilting point	v%	7	3	3
Field capacity	v%	15	8	3
Total available water	v%	8	5	3
Saturated hydraulic conductivity	cm/s	2.0 E-03	1.4 E-02	3
Organic matter	wt%	4.4 ± 0.7	1.2 ± 0.4	9
TOC	wt%	1.4 ± 0.3	0.1 ± 0.0	9
TKN	mg N/kg	1 510 ± 250	285 ± 145	5
NH ₄	mg N/kg	1.2 ± 0.5	0.8 ± 0.1	5
NOx	mg N/kg	2.4 ± 2.8	1.5 ± 0.5	5
Total P	mg P/kg	995 ± 140	667 ± 217	5
Available P	mg P/kg	86 ± 15	44 ± 30	9
Extractable Al	mg Al/kg	1 340 ± 80	1 160 ± 390	9
Extractable Fe	mg Fe/kg	275 ± 29	131 ± 35	9
Extractable Ca	mg Ca/kg	762 ± 111	253 ± 92	9
Extractable Mg	mg Mg/kg	87 ± 26	34 ± 15	9
Extractable K	mg K/kg	13 ± 10	24 ± 6	9

Table 1. Soil physical and chemical properties before experiment

Notes: n: number of samples randomly collected per depth, OM: organic matter; TOC: total organic carbon; NH₄: ammonia; NOx: nitrates plus nitrites.

2.3 Experimental setup and instrumentation

An experimental block design comprising three treatments, one groundwater loading rate (L0) and two primary effluent loading rates (L1 and L2) replicated three times for a total of nine 108 m² (10,8 x 10 m) experimental plots was set up (Figure 2). Plots were arranged from

north to south, from the lowest to the highest hydraulic loading (L0 to L2). This non-random plot layout was established to avoid groundwater contamination of low loading plots by high-loading plots as a result of groundwater flow (from north to south). L0 loading plots were irrigated with underground drinking water taken from an adjacent property located east of the plantation. Paddle-wheel flowmeters were installed upstream of each of these plots to monitor irrigated volumes of L0 plots. L1 and L2 loading plots were irrigated with a primary effluent of wastewater pumped from the WRRF. Screened wastewater taken from the city WRRF, was first settled into a septic tank equipped with a conventional septic tank filter, before being pumped to the willow plantation. A magnetic flowmeter was installed downstream of the septic tank to monitor irrigated volume of L1 and L2 plots.

A surface irrigation system was installed at the plantation prior to the experiment. Four rows per plot were simultaneously irrigated on a daily basis at an average irrigation rate of 10 mm/h (total irrigation area of 72 m² per plot). Irrigation took place during the day to maximize evapotranspiration. Irrigation sequence of plots was modified every two weeks to limit the effect of irrigation time of the day on results.

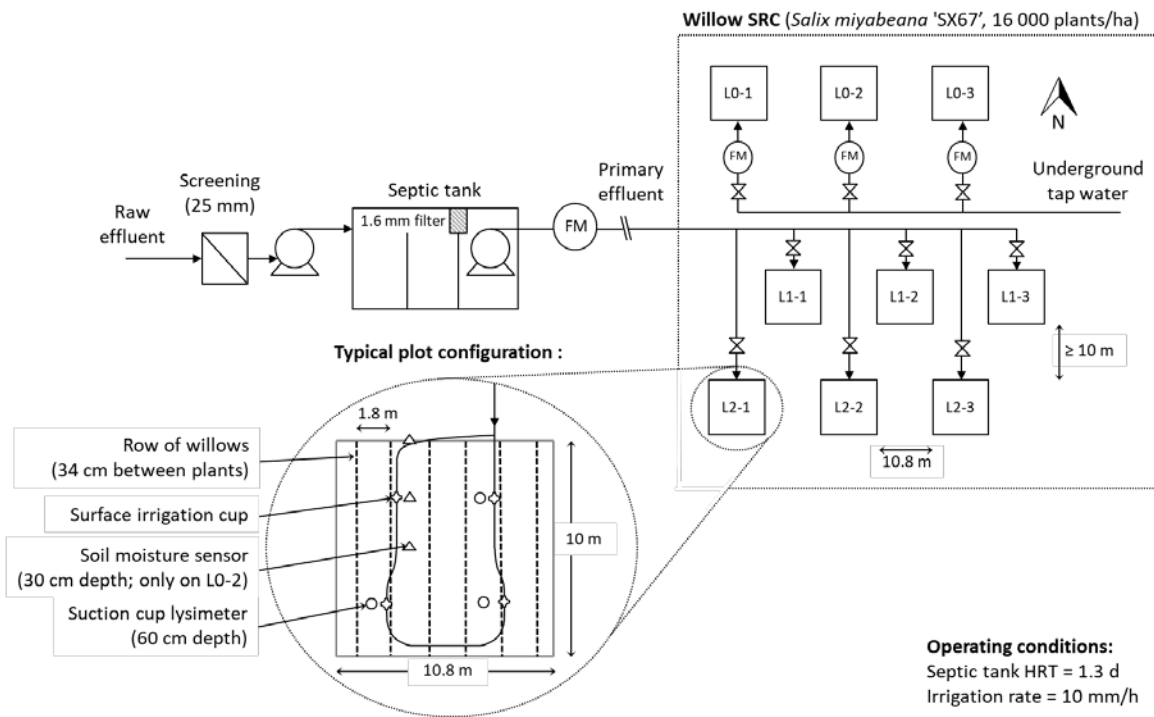


Figure 2. Experimental setup schematic. Notes: FM: flowmeter; HRT: hydraulic retention time; L: loading

The effect of irrigation on soil volumetric water content (VWC) was monitored with three dielectric volumetric soil moisture sensors (Decagon EC-5; 10 minute recording frequency) installed at a depth of 30 cm on one irrigated row of L0-2 plot. The manufacturer calibration equation was used, as recommended for mineral soils with electroconductivity (EC) of less than 8 dS/m (Decagon Devices, 2010). This calibration, which according to the manufacturer has an approximate accuracy of $\pm 3-4\%$ in this type of soil, was verified in the laboratory following the manufacturer procedure (Cobos and Chambers, 2010). The verification was carried out with one soil sample collected at the sensor depth of installation and on three VWCs ranging from 3 to 34 %. An average deviation of $2.2 \pm 1.0\%$, that was considered satisfactory, was obtained between VWC direct and sensor probe measures. Soil water filled

pore space (WFPS) was calculated by dividing VWC by soil porosity. Soil pore water quality was monitored with three suction cup lysimeters (Soilmoisture Equipment Corp. 1900L near-surface samplers) installed at a depth of 60 cm in each plot. Lysimeters were installed in June 2016 with bentonite clay and silica flour following the manufacturer instructions (Soilmoisture Equipment Corp., 2007). Meteorological parameters needed to estimate willow evapotranspiration (temperature, relative humidity, wind speed and solar radiation) were measured with a weather station installed at the southern end of the plantation (30 minute recording frequency). Meteorological data set was completed with L'Assomption weather station data according to the method suggested by Allen et al. (1998).

2.4 Sampling and analytical methods

Liquid sampling and analysis

Primary effluent and soil pore water samples were sampled every two weeks for a total of eight sampling campaigns. Primary effluent was sampled in the septic tank last compartment using an automatic sampler refrigerated with ice. One liter composite samples were collected over a 24-hour period at a rate of 42 mL/h. Soil pore water samples collected by the suction cup lysimeters were sampled in accordance with the manufacturer's guidelines (Soilmoisture Equipment Corp., 2007).

Total suspended solids (TSS) and volatile suspended solids (VSS) were measured according to APHA et al. (2012) using glass microfiber 1.2 μm filters (Whatman 934-AH, GE Healthcare Life Sciences). Chemical oxygen demand (COD) was measured according to APHA et al. (2012). Primary effluent biological oxygen demand (BOD_5) was estimated using the typical municipal wastewater ratio of COD to BOD_5 of 1.84 (EnviroSim, 2015).

Similarly, unbiodegradable soluble COD (S_U) was estimated with the typical wastewater ratio of S_U to COD of 0.08 (EnviroSim, 2015). Soil pore water biodegradable COD (COD_B) was estimated by subtracting primary effluent S_U to soil pore water COD. Soil pore water BOD_5 was estimated using a typical ratio COD_B to BOD_5 of 1.6 (EnviroSim, 2015). Nitrogen and phosphorus parameters were measured according to APHA et al. (2012) with a flow injection analysis system (Quickchem 8500, Lachat Instruments). Total nitrogen (TN) was calculated by summing TKN and NO_x . pH and EC were measured with a pH meter (SevenEasy, Mettler Toledo) and a conductivity meter (SevenCompact, Mettler Toledo). Ca, Mg, K, Na, and SO_4 were measured with an atomic absorption spectroscopy instrument (AAnalyst 200, Perkin Elmer). Cl was measured with chloride test strips (Quantab CAT 27449-40, HACH). Analytical results below the detection limit of analytical methods were considered to be equal to the limit of detection (Croghan and Egeghy, 2003).

Soil sampling and analysis

Soil samples were taken 8 and 10 days after the end of the experiment on November 16 and 18, 2016. Samples were taken at depths of 20 ± 3 and 50 ± 3 cm near the four irrigation outlets of each plot (total of 72 samples, 36 per depth). Soil composite samples were taken in three holes drilled within one meter of irrigation cups.

Organic matter content (OM) was measured with the loss on ignition method (ASTM International, 2014). Total organic carbon (TOC) was measured by LECO Combustion Analysis (combustion and infrared carbon dioxide detection) after HCl pretreatment (LECO CS 744, LECO Corporation). NH_4 and NO_x were extracted by KCl (CEAEQ, 2015) and analyzed with a flow injection analysis system (Quickchem 8500,

Lachat Instruments). Soil available P was extracted by the Mehlich 3 method (Mehlich, 1984) according to CEAEQ (2014) and measured using the ascorbic acid molybdate blue method (Denning et al., 2011) with a spectrophotometer at 880 nm (DR6000, HACH). Soil pH was measured in a soil-salt (1:2 CaCl₂) solution (Burt & Soil Survey Staff, 2014) with a pH meter (SevenEasy, Mettler Toledo). Soil EC was measured with the 1:1 soil-to-water method (Denning et al. 2011) using a conductivity meter (SevenCompact, Mettler Toledo). Soil exchangeable Al, Fe, Ca, Mg and K were extracted by the Mehlich 3 method (Mehlich, 1984) according to CEAEQ (2014) and measured with an atomic absorption spectroscopy instrument (AAnalyst 200, Perkin Elmer).

Biomass sampling and analysis

Aboveground woody biomass was sampled two weeks after the end of the experiment (November 22, 2016). Five plants per plots were randomly selected, cut directly above ground level and weighted on site. Segments from the bottom, middle and top of stems were collected on each plant samples to estimate dry biomass yield. Uptake of nitrogen and phosphorus in woody biomass was estimated using the typical respective contents of 0,4 % to 1,0 % and 0,04 % to 0,10 % respectively for these nutrients in willow wood following the irrigation of wastewater or biosolids (Dimitriou and Aronsson, 2004; Labrecque and Teodorescu, 2003; Labrecque et al., 1998; Larsson et al., 2003). Uptake of nitrogen in root and leaf biomass plus denitrification was estimated by subtracting uptake in woody biomass from total nitrogen applied during the experiment. Uptake of nitrogen in root and leaf biomass (temporary N removal due to recycling following leaves and roots decomposition) was not differentiated from denitrification (permanent N removal) because of the high

uncertainties associated with estimating these two components of the nitrogen cycle. In a similar way, uptake of phosphorus in root and leaf biomass plus soil adsorption and precipitation was estimated jointly by subtracting uptake in woody biomass from total phosphorus applied.

2.5 Water balance and contaminant loading estimations

Crop evapotranspiration (ET_c) was estimated using the FAO crop coefficient approach, which consists of multiplying reference evapotranspiration (ET_o) to a crop coefficient (k_c) (Allen et al., 1998). Unfertilized and fertilized one-year-old willow crop coefficients obtained from Guidi et al. (2008) were used to estimate L0 and L1 and L2 plots ET_c , respectively. ET_c of the plots were adjusted to account for water shortage in the root zone reserve corresponding to the first 30 centimeters of soil. Effect of soil salinity on ET_c was neglected since soil EC measured in the root zone of L1 and L2 plots after the experiment (0.42 dS/m) was well below the critical threshold from which salinity is considered to have an impact on willow transpiration (5.0 dS/m; Hangs et al., 2011). Deep percolation out of the root zone was estimated for each plot on a daily basis according to the FAO method (Allen et al., 1998) using ET_c estimated values and rainfall and irrigation measured volumes.

For each sampling period, organic, nitrogen and phosphorus loadings applied on each plot were estimated by multiplying the primary effluent concentrations to the total irrigation volume applied during the two weeks preceding sampling. Similarly, contaminants loadings out of the root zone of each plot were estimated by multiplying the average soil pore water concentrations by the total deep percolation volume for the two-week period preceding sampling. That is, concentrations were considered constant at the influent and the effluent of

each plot over the two-week periods preceding each sampling campaign (i.e. no concentration interpolation between sampling).

2.6 Irrigation treatments

Experimental plots were irrigated under one groundwater loading rate ($L_0 = 14 \pm 0$ mm/d, total of 1 510 mm) and two primary effluent loading rates ($L_1 = 10 \pm 5$ mm/d and $L_2 = 16 \pm 7$ mm/d, totals of 1 160 mm and 1 820 mm, respectively) for 111 days (Table 2). These hydraulic loadings, when expressed on a seasonal basis of 180 days, fall in the hydraulic loading rate (HLR) range of 500 to 6 000 mm/yr recommended by the US EPA (2006) for slow-rate land application systems.

The primary municipal effluent irrigated had an average concentration of 224 mg COD/L, 122 mg BOD₅/L, 31 mg N/L, 3.1 mg TP/L and 1.5 mg o-PO₄/L and a pH of 7.7 (Table 3). These concentrations corresponded to total applied loadings of 2 650 kg COD/ha, 1 440 kg BOD₅/ha, 370 kg N/ha, 37 kg P/ha and 19 kg P/ha for the L1 loading and 4 150 kg COD/ha, 2 255 kg BOD₅/ha, 580 kg N/ha, 58 kg P/ha and 29 kg P/ha for the L2 loading. Nitrogen content was mainly measured in its ammonia form and a few times in its TKN form. The primary effluent was characterized by average BOD₅ to TKN, BOD₅ to TP and TKN to TP and K (N:P:K) ratios of 4.0, 38 and 100:10:13, respectively.

Parameter	L0		L1		L2	
	mm	mm/d	mm	mm/d	mm	mm/d
Rainfall	389	4 ± 10	389	4 ± 10	389	4 ± 10
Irrigation	1 510	14 ± 0	1 160	10 ± 5	1 820	16 ± 7
ET _c	270	2 ± 2	470	4 ± 4	510	5 ± 4
DP	1 630	15 ± 10	1 080	10 ± 11	1 690	15 ± 12

Table 2. Total and average daily rainfall, irrigation, estimated crop evapotranspiration (ET_c) and estimated deep percolation out of the root zone (DP) during experiment

Parameters	Units	Average values	n	Typical values ¹
COD	mg COD/L	224 ± 53	8	339 / 508 / 1 016
S _U	mg COD/L	18 ± 4	8	
BOD ₅	mg O ₂ /L	118 ± 28	8	133 / 200 / 400
TSS	mg/L	54 ± 16	8	130 / 195 / 389
VSS	mg/L	46 ± 16	8	
FSS	mg/L	8 ± 2	8	
TN	mg N/L	31 ± 8	8	23 / 35 / 69
TKN	mg N/L	31 ± 8	8	24 / 34 / 70
N _{org}	mg N/L	9 ± 5	7	10 / 14 / 29
NH ₄	mg N/L	20 ± 3	7	14 / 20 / 41
NO _x	mg N/L	0.05 ± 0.03	7	0 / 0 / 0
TP	mg P/L	3.1 ± 0.6	8	3.7 / 5.6 / 11.0
o-PO ₄	mg P/L	1.5 ± 0.5	7	
pH	--	7.7 ± 0.2	8	
EC	dS/m	2.7 ± 1.4	8	
Ca	mg Ca/L	142 ± 83	5	
Mg	mg Mg/L	41 ± 19	5	
K	mg K/L	13 ± 4	5	11 / 16 / 32
Na	mg Na/L	371 ± 192	4	
SO ₄	mg S/L	34 ± 8	5	8 / 12 / 24 ²
Cl	mg Cl/L	628 ± 528	3	39 / 59 / 118 ²

Table 3. Primary effluent characterization. Note: S_U: Estimated soluble unbiodegradable COD

Notes: ¹ Typical low / medium / high strength concentrations of untreated domestic wastewater (M&EA, 2014) ² Typical values without potable water background concentration

2.7 Statistical analysis

One-way ANOVA and Tukey HSD post-hoc analysis were performed on soil and biomass data to assess whether irrigation loadings had a significant effect on soil characteristics and biomass yields. The model comprised three blocks as a random factor (block 1: L0-1, L1-1 and L2-1; block 2: L0-2, L1-2 and L2-2; block 3: L0-3, L1-3 and L2-3) and irrigation loadings as a fixed factor. A significance level of 5 % was considered for all analyses. Normal distribution of residuals was assessed visually with quantile-quantile plots. A log transformation (base-10) was performed on the datasets that presented a skewed distribution of their residuals (OM, TOC, Ca, Mg, pH and EC at a depth of 20 cm and NO_x and K at both 20 and 50 cm). Since a significant number of NH₄ results were measured at the analytical limit of detection (> 85% of samples for both 20 and 50 cm), only descriptive statistics are presented for this parameter. Analyses were carried out with R software (nmle and multcomp modules).

3. Results

3.1 Soil water content

A daily average soil WFPS of 65 ± 17 % was measured at the L0-2 plot during the first 13 weeks of the irrigation period which increased to an average value of 88 ± 7 % during the last three weeks of the experiment (Figure 3). The heavy rains measured in mid-October seem to have caused this increase in soil moisture content (Figure 1). Similar rainfall events that

occurred in mid-August did not have such an effect on soil WFPS. The higher rate of plant transpiration and soil evaporation at this period of summer probably explains this lower effect of rainfall on soil water content. Soil WFPS reached a level above 60%, which corresponds to the minimum threshold required for soil denitrifying activity (Havlin et al., 2013), for an average duration of 1 h/d during the first thirteen weeks of the experiment and an average duration of 24 h/d during the last three. Favorable soil moisture conditions favorable to soil denitrification, i.e. a WFPS greater than 80%, were not reached during the first thirteen weeks, but were reached for an average duration of 19 h/d during the last three.

Prolonged surface ponding was observed on the westernmost plots of the experimental setup (L0-1, L1-1 and L2-1) during these last three weeks. Some rows crossing these plots contained agricultural machinery ruts and a more heavily compacted soil (qualitative observation). The lower hydraulic conductivity presumably associated to this higher soil compaction, in addition to lower plant transpiration and higher rainfall at this period of the year, could have caused the reported water surface accumulation. Soil compaction and infiltration capacity, in addition to soil texture, will need to be considered to design SRWC vegetation filters. These observations suggest that irrigation loads may have exceeded willow water requirements, especially during the last three weeks of the experiment, and indicate an imbalance between the constant wastewater HLR applied during the experiment and willow seasonal water requirement.

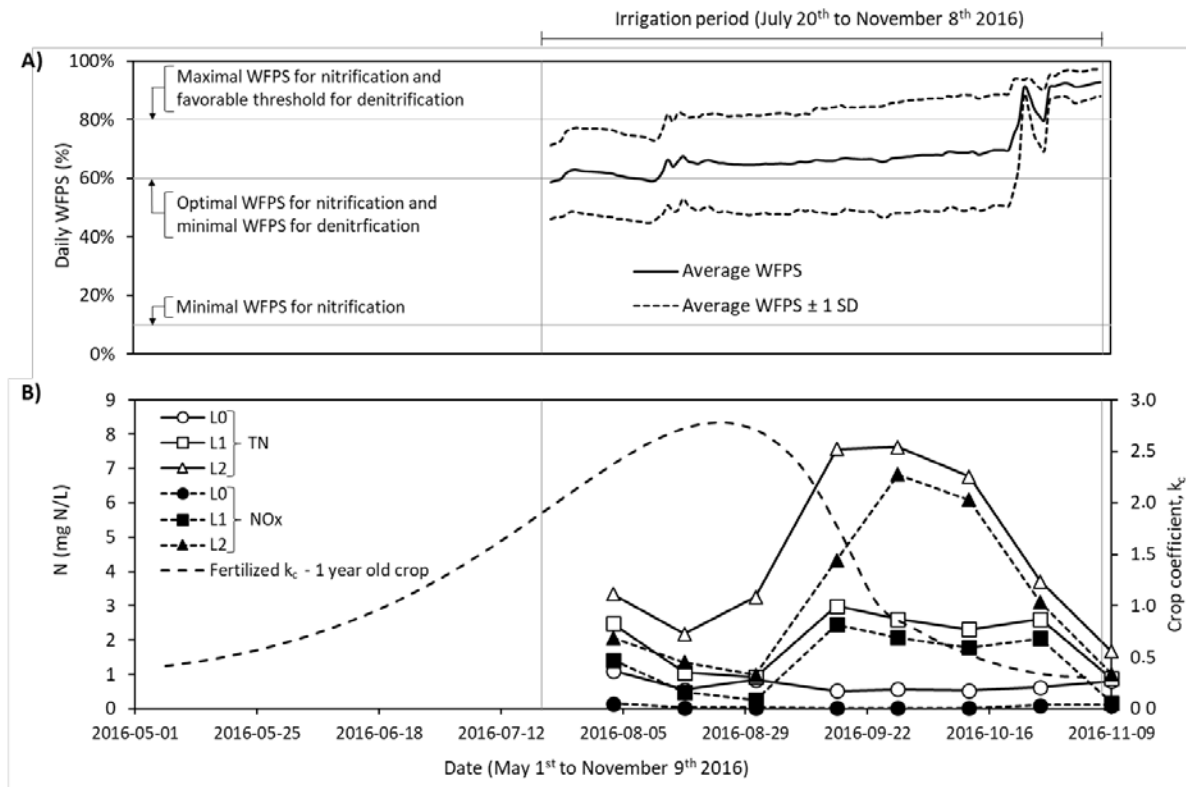


Figure 3. Nitrogen (N) removal conditions: A) Percentage daily water filled pore space (WFPS) and nitrification and denitrification WFPS requirements (adapted from Havlin et al., 2013 and Paul, 2007); B) Total nitrogen (TN) and nitrites and nitrates (NOx) measured in water collected by the lysimeters and fertilized crop coefficient of one-year-old willows (k_c ; adapted from Guidi et al., 2008)

3.2 Removal efficiency

Organic matter

Similar average COD load removal efficiency of $91 \pm 6\%$ and $91 \pm 4\%$ were achieved for L1 and L2 loadings (Table 4). Similar average soil pore water COD concentrations of 18 ± 6 mg/L, 21 ± 9 mg/L and 22 ± 8 mg/L were also measured for the L0, L1 and L2 loadings, respectively. A decrease of L0 soil pore water COD was observed throughout the experiment which may indicate a soil background soluble organic matter leaching out of the root zone.

L1 and L2 COD concentrations remained stable and were not influenced by the primary effluent COD concentration variations all along the experiment (primary effluent average COD of 224 ± 53 mg/L). L1 and L2 COD concentrations were similar to those of L0 and remained close to the estimated primary effluent S_U throughout the irrigation period (Figure 4). These results suggest an essentially complete oxidation of the primary effluent biodegradable organic matter content.

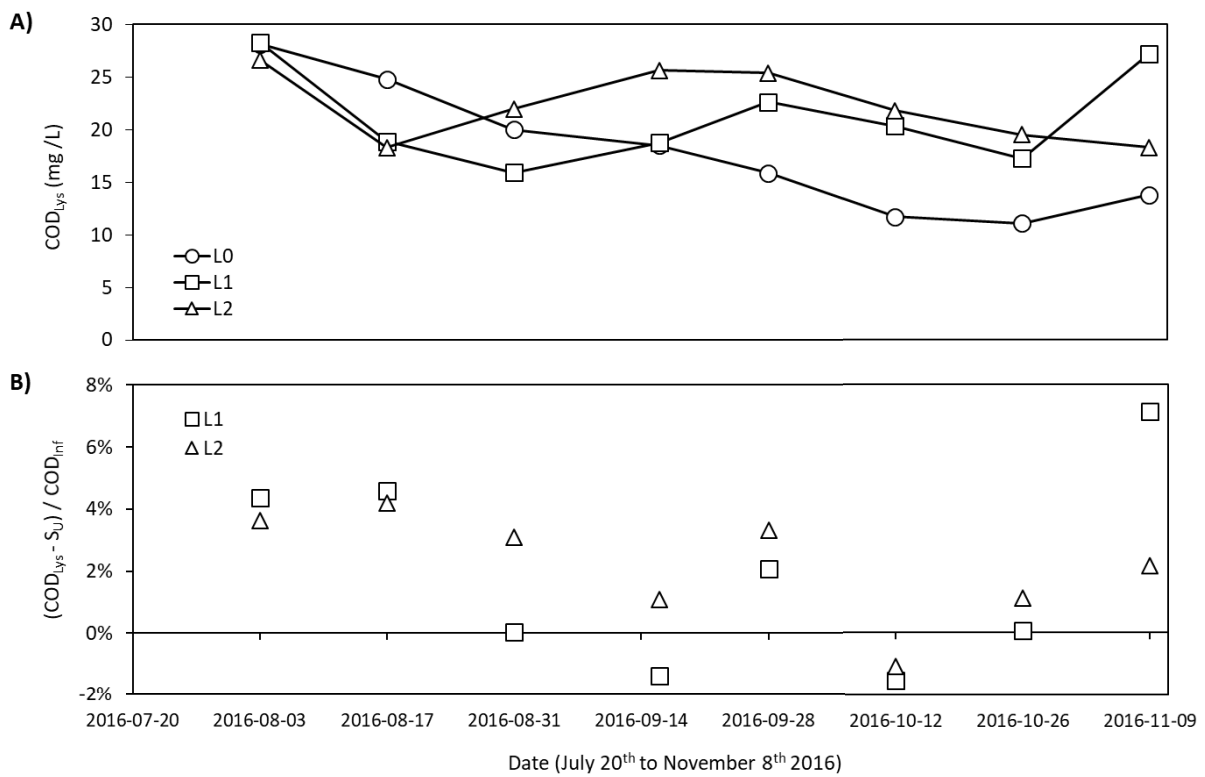


Figure 4. Remaining organic matter in collected water by the lysimeters over the course of the experiment: A) COD; B) Estimated biodegradable soluble fraction of COD ($((\text{COD}_{\text{Lys}} - S_U) / \text{COD}_{\text{Inf}})$)

Nitrogen

As illustrated at Figure 5, average TKN load removal efficiency of $98 \pm 1\%$ and $98 \pm 6\%$ were achieved for L1 and L2 loadings, respectively. Average soil pore water TKN

concentrations of 0.6 ± 0.3 mg/L, 0.7 ± 0.2 mg/L and 1.3 ± 2.2 mg/L were measured for the L0, L1 and L2 loadings, respectively. L0 and L1 TKN concentration remained stable all along the experiment. The higher standard deviation obtained for L2 soil pore water concentration was partly caused by higher NH_4 concentrations measured at two lysimeters of the L2-2 plot at the sixth and eighth weeks of the experiment (8.1 and 8.4 mg N/L at week 6 (August 31, 2016) and 9.8 and 12.8 mg/L at week 8 (September 14, 2016)). These higher concentrations could have been caused by a temporary accumulation of NH_4 induced by the degradation of an external source of organic nitrogen around this plot (Kadlec and Wallace, 2009). Apart from this event of higher concentrations, the results suggest an almost complete removal of wastewater ammonia.

Average TN load removal efficiency of $94 \pm 11\%$ and $87 \pm 17\%$ were achieved for L1 and L2 loadings, respectively (Table 4). Average soil pore water TN concentrations of 0.7 ± 0.3 mg/L, 2.0 ± 2.7 mg/L and 4.5 ± 6.0 mg/L were observed for the L0, L1 and L2 loadings, respectively. Soil pore water nitrogen measured at the L0 plot was mainly in the organic form, whereas nitrogen at L1 and L2 was mainly measured as nitrites and nitrates.

The L0 plot soil pore water TN concentration remained stable during the experiment, while L1 and L2 concentrations increased in early September, which corresponds to the willows transpiration decline period (Figure 3). The total nitrogen concentration measured in L1 and L2 plots then remained to a relatively high level from early-September to mid-October before falling back to a level similar to that at the start of the experiment. The decrease in TN concentration observed at the end of the experiment, especially for the L2 loading, occurred during the high soil WFPS period previously discussed.

Parameter	Loading	Influent			Lysimeter			Removal efficiency	
		Average concentration mg/L	Total loading kg/ha	Average daily loading mg m ⁻² d ⁻¹	Average concentration mg/L	Total loading kg/ha	Average daily loading mg m ⁻² d ⁻¹	Concentration %	Loading %
COD	L0				18 ± 12	263 ± 116	230 ± 170		
	L1	224 ± 53	2 650 ± 230	2 400 ± 1 000	21 ± 9	205 ± 20	180 ± 110	90 ± 5	91 ± 6
	L2	224 ± 53	4 150 ± 320	3 700 ± 1 500	22 ± 8	362 ± 37	320 ± 130	90 ± 4	91 ± 4
BOD ₅	L0								
	L1	122 ± 29	1 440 ± 125	1 300 ± 500	2.7 ± 5.6	23 ± 17	20 ± 50	97 ± 6	98 ± 6
	L2	122 ± 29	2 255 ± 180	2 000 ± 800	2.9 ± 4.0	44 ± 19	40 ± 60	97 ± 4	98 ± 3
TN	L0				0.7 ± 0.3	10 ± 0.4	10 ± 3		
	L1	31 ± 8	370 ± 36	300 ± 100	2.0 ± 2.7	22 ± 13	20 ± 30	93 ± 9	94 ± 11
	L2	31 ± 8	580 ± 43	500 ± 200	4.5 ± 6.0	73 ± 47	70 ± 90	85 ± 19	87 ± 17
NH ₄	L0				0.05 ± 0.02	0.8 ± 0.2	0.75 ± 0.29		
	L1	22 ± 4	254 ± 25	200 ± 100	0.05 ± 0.01	0.5 ± 0.05	0.45 ± 0.22		
	L2	22 ± 4	401 ± 29	400 ± 100	0.63 ± 2.21	8.7 ± 12.7	10 ± 30		
NO _x	L0				0.02 ± 0.07	0.9 ± 0.1	0.83 ± 0.87		
	L1	0.05 ± 0.03	0.5 ± 0.0	0.6 ± 0.2	1.34 ± 2.69	16 ± 13	10 ± 30		
	L2	0.05 ± 0.03	0.7 ± 0.0	0.8 ± 0.4	3.22 ± 5.26	54 ± 41	50 ± 80		
TP	L0				0.08 ± 0.03	1.3 ± 0.2	1.1 ± 0.56		
	L1	3.1 ± 0.6	37 ± 3	30 ± 10	0.07 ± 0.02	0.7 ± 0.02	0.61 ± 0.41	98 ± 0,1	98 ± 0.1
	L2	3.1 ± 0.6	58 ± 4	50 ± 20	0.08 ± 0.03	1.2 ± 0.2	1.1 ± 0.45	98 ± 0.1	98 ± 0.1
PO ₄ ^{o-}	L0				0.02 ± 0.02	0.3 ± 0.02	0.25 ± 0.18		
	L1	1.5 ± 0.5	19 ± 2	20 ± 10	0.02 ± 0.02	0.1 ± 0.07	0.13 ± 0.21		
	L2	1.5 ± 0.5	29 ± 2	30 ± 10	0.01 ± 0.01	0.2 ± 0.03	0.22 ± 0.18		
pH	L0				7.0 ± 0.3				
	L1	7.7 ± 0.2			7.0 ± 0.3				
	L2	7.7 ± 0.2			6.9 ± 0.4				
EC	L0				0.4 ± 0.2				
	L1	2.7 ± 1.4			2.1 ± 0.7				
	L2	2.7 ± 1.4			2.2 ± 0.2				

Table 4. Average values of concentration and estimated loading at the influent and lysimeters and concentration and estimated loading removal efficiency

Nitrogen woody biomass uptake ranging from 88 to 220 kg N/ha and 104 to 260 kg N/ha were estimated for L1 and L2, respectively (Figure 5). These uptake account for 24% to 59% and 18 to 45% of L1 and L2 nitrogen applied during the experiment, respectively. Uptake in root and leaf biomass plus denitrification of 128 to 260 kg N/ha and 247 to 403 kg N/ha for L1 and L2, which account for 35 % to 70 % and 43 % to 69 % of nitrogen applied, were also estimated for L1 and L2, respectively. Although presenting uncertainties, these estimations suggest that both denitrification and willow uptake played a significant role in nitrogen removal.

Phosphorus

Average TP load removal efficiency of $98 \pm 0.1\%$ was achieved for both L1 and L2 loadings. Average soil pore water TP concentrations of 0.08 ± 0.03 , 0.07 ± 0.02 and 0.08 ± 0.03 mg P/L and o-PO₄ concentrations of 0.02 ± 0.02 , 0.02 ± 0.02 and 0.01 ± 0.01 mg P/L were measured for the L0, L1 and L2 loadings, respectively. L1 and L2 TP and o-PO₄ concentrations remained very close to those of L0, and thus to the soil background concentration, throughout the irrigation period, which suggests a complete removal of wastewater phosphorus.

Phosphorus woody biomass uptake ranging from 9 to 22 kg P/ha and 10 to 26 kg P/ha were estimated for L1 and L2, respectively (Figure 5). These uptakes account for 24% to 59% and 18 to 45% of L1 and L2 TP applied during the experiment, respectively. Accumulation in root and leaf biomass plus soil adsorption and precipitation of 14 to 28 kg P/ha and 31 to 46 kg P/ha for L1 and L2, which account for 39 % to 74 % and 53 % to 80 % of TP applied, were also estimated for L1 and L2, respectively. As for total nitrogen and although presenting some uncertainties, these estimations suggest that both willow uptake and soil adsorption and precipitation played a significant role in phosphorus removal.

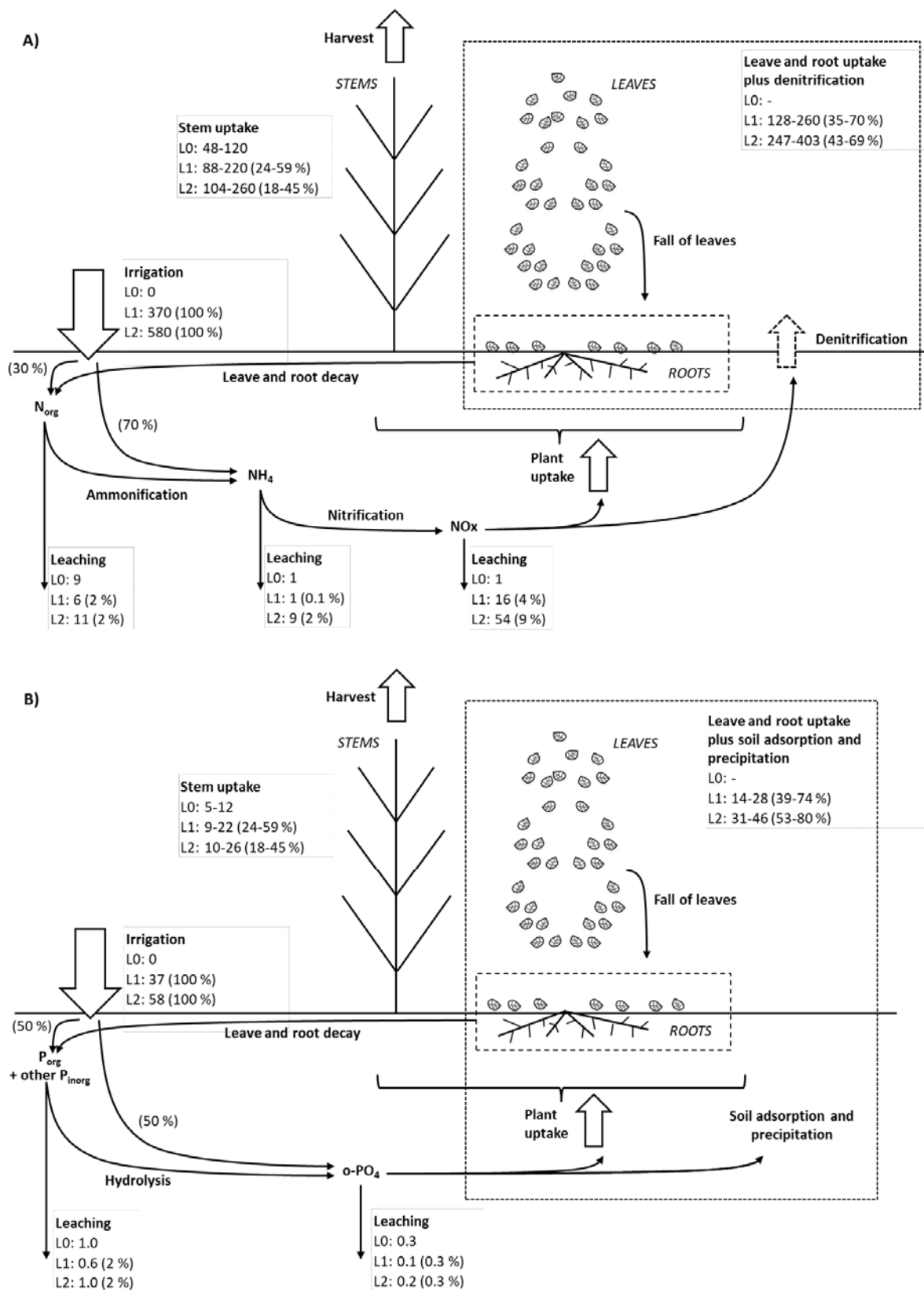


Figure 5. Quantities (kg/ha) of A) N and B) P applied over the course of the experiment and estimated fate after irrigation (proportions of loadings applied are presented in parentheses).

Electrical conductivity

Average soil pore water EC of 0.4 ± 0.2 , 2.1 ± 0.7 and 2.2 ± 0.2 dS/m were measured for the L0, L1 and L2 loadings, respectively (Table 4). The difference between potable and wastewater irrigated plots EC indicates that samples taken from the plots irrigated with the primary effluent were indeed the result of wastewater percolation through soil.

3.3 Effect on soil chemical properties

Irrigation at L1 and L2 wastewater loadings resulted in a significant change in some soil chemical properties (NO_x, Fe, Mg and EC) at a depth of 20 cm as compared to L0 with potable water loading (Table 5). For this same depth, no significant differences in soil properties were observed between the L1 and L2 treatments, except for calcium, where the L2 treatment led to an increase of calcium soil content. No strong significant differences in soil chemical properties were observed at a depth of 50 cm following the three treatments, except for iron and EC, where the irrigation at L1 and L2 loadings led to a decrease of soil iron content and an increase of soil EC as compared to L0.

The amendment of organic matter induced by the application at L1 and L2 loadings (totaling 2 650 and 4 150 kg COD/ha, respectively) did not have a significant effect on soil OM or TOC contents at 20 and 50 cm as compared to the potable water irrigated soil sampled results. This indicates that the soil at the experimental site has a high OM mineralization capacity and is consistent with the reported high COD and BOD₅ removal efficiency. Irrigation at the two primary effluent loadings did not seem to increase soil NH₄ content but resulted in a significant increase of NO_x content at 20 cm. Although probably temporary, this increase in soil NO_x content is an indication of soil nitrification activity. Irrigation at the L2 loading resulted in a significant increase in soil available phosphorus content at 20 cm, which suggests an eventual phosphorus soil saturation should wastewater irrigation be continued over a number of years.

Depth Loading	20 cm				50 cm			
	L0	L1	L2	<i>p</i> value	L0	L1	L2	<i>p</i> value
Parameter								
OM (wt%)	3.5 ± 0.2 a	3.7 ± 0.2 a	3.6 ± 0.2 a	0.191	1.2 ± 0.3 a	1.5 ± 0.6 a	1.5 ± 0.4 a	0.117
TOC (wt%)	1 ± 0.1 a	1.1 ± 0.1 a	1 ± 0.1 a	0.109	0.1 ± 0.0 a	0.2 ± 0.1 a	0.2 ± 0.1 a	0.168
NH ₄ (mg N/kg)	0.6 ± 0.1	0.5 ± 0.02	0.5 ± 3E-04		0.5 ± 0.02	0.5 ± 3E-04	0.6 ± 0.1	
NO _x (mg N/kg)	0.1 ± 6E-05 a	2.7 ± 1.5 b	2.7 ± 1.2 b	<.0001*	0.1 ± 8E-05 a	0.6 ± 0.5 a	1.4 ± 1.7a	0.168
Available P (mg P/kg)	87 ± 9 a	89 ± 6 ab	96 ± 8 b	0.004*	37 ± 8 a	41 ± 16 a	50 ± 16 a	0.098
Al (mg Al/kg)	1 280 ± 90 a	1 210 ± 110 a	1 170 ± 150 a	0.59	1 210 ± 240 a	1 040 ± 160 a	1 200 ± 160 a	0.142
Fe (mg Fe/kg)	330 ± 34 a	285 ± 24 b	282 ± 23 b	0.0004*	181 ± 42 a	141 ± 32 b	122 ± 18 b	0.002*
Ca (mg Ca/kg)	549 ± 76 a	581 ± 77 a	731 ± 71 b	0.0002*	232 ± 62 a	291 ± 79 a	271 ± 59 a	0.13
Mg (mg Mg/kg)	72 ± 14 a	96 ± 25 b	95 ± 20 b	0.034*	28 ± 10 a	44 ± 21 b	38 ± 11 ab	0.0406*
K (mg K/kg)	36 ± 11 a	30 ± 9 a	39 ± 9 a	0.2233	27 ± 12 a	16 ± 6 b	18 ± 4 ab	0.02*
pH	5.6 ± 0.1 a	5.7 ± 0.1 ab	5.8 ± 0.1 b	0.008*	5.6 ± 0.2 a	5.8 ± 0.3 a	5.8 ± 0.1 a	0.059
EC (dS/m)	0.1 ± 0.01 a	0.42 ± 0.05 b	0.42 ± 0.06 b	<.0001*	0.06 ± 0.01 a	0.38 ± 0.09 b	0.46 ± 0.04 b	<.0001*

Different letters indicate a significant difference with $p < 0.05$.

Table 5. Chemical soil properties (mean ± standard deviation, SD) following the irrigation with three different loadings at the end of the experiment and results of one-way Anova tests.

Irrigation at the L1 and L2 loadings had no significant effect on soil extractable aluminum content but resulted in a decrease in extractable iron content at 20 cm and 50 cm, which suggests that soil wastewater percolation resulted in the formation of iron minerals which are more difficult to extract. The irrigation at the L2 loading resulted in a significant increase in extractable calcium at 20 cm as compared to the L1 and L0 loadings. The application of a primary effluent resulted in a significant increase in soil EC at 20 cm and 50 cm, which is consistent with the highest EC measured in L1 and L2 plots soil pore water throughout the experiment.

3.4 Biomass yield

Average annual biomass yields of 12 ± 4 , 22 ± 8 and 26 ± 7 t DM ha⁻¹ yr⁻¹ were measured in the L0, L1 and L2 treatments, respectively. The irrigation at the L1 and L2 loadings resulted in a significant increase in woody biomass yield as compared to potable water irrigation (p value < 0.0001). The highest fertilizer load of the L2 loading did not result, however, in a significant increase in biomass yield as compared to the L1 loading. This indicates that the fertilizing load associated with the L2 loading exceeded the willow nutritional requirements and that the maximal amount of recuperated resources was reached at the L1 treatment.

4. Discussion

4.1 Water balance

Soil water content

The average daily HLR of L0, L1 and L2 were well below the maximum rate of 73 mm/d typically recommended for soils with a hydraulic conductivity similar to the one of the experimental site, i.e. 2.0×10^{-3} cm/s (Crites et al., 2006). This suggests that soil infiltration capacity is not the limiting design parameter of SRWCs operated on well drained sandy-textured soils. Soil water content results, however, indicated an imbalance between the constant rate irrigation applied during the

experiment and the water requirements of willows, resulting in a water surplus in periods of low plant transpiration and heavy autumn rainfall. A modulation of irrigation according to the typical seasonal patterns of willow transpiration would allow a better distribution of the irrigated effluent with regard to the needs of the willows. The use of soil moisture probes could help implement such flow modulation.

Evapotranspiration

The estimated average ET_c rates of 4 and 5 mm/d for L1 and L2 (Table 2) are in the same order of magnitude, if somewhat slightly lower, than those reported in the literature following the application of municipal wastewater. The ET_c rates reported are lower than the average value of 7 to 8 mm/d reported by Dimitriou and Aronsson (2011) following the application of loadings of 316 kg N ha⁻¹ yr⁻¹ and 26 kg P ha⁻¹ yr⁻¹ similar to those of the L1 treatment (Table 4). The estimated ET_c are also slightly lower to the average rate of 4 to 7 mm/d reported by Curneen and Gill (2014) following the irrigation with a primary effluent, in which lower N and P loadings were applied (116-147 kg N ha⁻¹ yr⁻¹ and 12 kg P ha⁻¹ yr⁻¹).

The reported ET_c values are also slightly lower to ET_c rates reported in the literature following the application of biosolids: 7 to 9 mm/d (Dimitriou and Aronsson, 2004), 3 mm/d (Dimitriou and Aronsson, 2011), landfill leachate: 2 to 8 mm/d (Dimitriou and Aronsson, 2010) and synthetic fertilizer: 3 to 8 mm/d (Aronsson and Bergstrom, 2001), 7 to 8 mm/d (Dimitriou and Aronsson, 2004), 7 to 12 mm/d (Guidi et al., 2007) and 5 to 7 mm/d (Pistocchi et al., 2009). Moreover, a recent study published by Frédette et al. (2019) exhibit the high evapotranspiration potential of *Salix miyabeana* 'SX67' grown under humid continental climate, with reported average seasonal ET_c of 16.8 mm/d. Although this study was conducted under very favorable conditions for evapotranspiration (high water availability and oasis and clothesline effects), the high ET_c rates reported suggests that the present study values may have been underestimated.

The ET_c estimation method used in this study presents some limitations that could explain such an underestimation. Willow crop coefficients used to estimate ET_c were obtained by Guidi et al. (2008) as part of an experiment carried out with a different willow species, *Salix alba*, and under Mediterranean climate. Moreover, these k_c were derived from first-year willow shoots of one-year-old plants, while the present study was carried out with first-year shoots of eight-year-old plants. This difference in crop age, and most likely in root system establishment degree, support the assumption that the use of Guidi et al. (2008) coefficients may have led to an underestimation of ET_c rates. Such an underestimation would have led to an overestimation of deep percolation volume out of the root zone and thus, to an overestimation of the pollutant loadings at the lysimeters and an underestimation of the loading removal efficiencies. This represents a conservative limitation of this study.

4.2 Removal efficiency

Organic matter

The high COD ($91 \pm 6\%$ and $91 \pm 4\%$) and estimated BOD₅ ($98 \pm 6\%$ and $98 \pm 3\%$) load removal efficiencies achieved for L1 and L2 showed a high organic matter removal capacity for SRWC vegetation filters operated in coarse soil.

Several factors can explain the almost complete organic matter removal achieved over the duration of the experiment. First, L1 and L2 average BOD₅ loadings (1.3 and $2.0 \text{ kg ha}^{-1} \text{ d}^{-1}$) were well below the lower bound of daily organic loading rate of 50 to $500 \text{ kg BOD}_5 \text{ ha}^{-1} \text{ d}^{-1}$ typically recommended for slow infiltration land application systems (US EPA, 2006). These low loading rates were caused by the low primary effluent COD and BOD₅ concentrations, which were below the concentrations associated to a typical low-strength domestic effluent (Table 3). Furthermore, the coarse texture soil of the experimental site was, by nature, favorable to soil aeration and organic matter oxidation (Veen and Kuikman, 1990). In addition, the resting periods between daily

wastewater applications maintained during the experiment (average of 22.9 and 22.4 hours for L1 and L2, respectively) most likely induced a WSPS favorable to aerobic oxidation and organic matter removal (Figure 3). Accordingly, the average WSPS of $65 \pm 17 \%$ measured at the L0-2 plot during the first thirteen weeks of the experiment falls within the 50-80% WSPS beneficial to heterotrophic bacteria activity (Havlin et al., 2013). The increase in WSPS during the last three weeks of the experience at an average level of $88 \pm 7 \%$ was not reflected in a decrease in COD removal efficiency.

The organic removal efficiency achieved are consistent with the high removal efficiency typically associated with slow infiltration systems (US EPA, 2006). The average COD concentration removal efficiencies observed for L1 ($90 \pm 5 \%$) and L2 ($90 \pm 4 \%$) are similar that of 85 % reported by Miguel et al. (2014) following the irrigation of a poplar plantation with a primary effluent at an organic loading rate of $486 \text{ kg BOD}_5 \text{ ha}^{-1} \text{ yr}^{-1}$. Similarly, the BOD_5 concentration removal efficiencies estimated for L1 ($96 \pm 6 \%$) and L2 ($96 \pm 4 \%$) are similar to that of 94 % reported by Perttu and Kowalik (1997) following an organic loading rate of $3\,900 \text{ kg BOD}_5 \text{ ha}^{-1} \text{ yr}^{-1}$ (Table 6). The lowest organic removal efficiencies reported by Larsson et al. (2003), following organic loading rates ranging from 558 to $1\,506 \text{ kg BOD}_5 \text{ ha}^{-1} \text{ yr}^{-1}$, could be explained by the fine texture and organic nature of their soil, which may have been unfavorable to aerobic soil activity.

There are currently no BOD_5 nor COD standards in Québec for municipal wastewater treatment systems that discharge their effluent by soil infiltration. The results obtained were therefore compared to the municipal surface water discharge standard of $25 \text{ mg BOD}_5/\text{L}$ for (Gouvernement du Québec, 2017). The average BOD_5 concentrations of soil pore water estimated for L1 and L2 (2.7 ± 5.6 and $2.9 \pm 4.0 \text{ mg BOD}_5/\text{L}$) are below this standard. These concentrations are also below the Québec standard of $15 \text{ mg BOD}_5/\text{L}$ required for an advanced secondary treatment from an isolated residence (MDDELCC, 2015b).

Effluent	Effluent N:P:K ratio	Average HLR mm / yr	Total loading			Average concentration / Loading removal efficiency			Soil top layer texture	Willow variety	Average biomass yield t DM ha ⁻¹ yr ⁻¹	Reference
			BOD ₅	TN	TP	BOD ₅	TN	TP				
			kg ha ⁻¹ yr ⁻¹			%						
Untreated	100:8: -	910	-	316	26	- / -	- / 96	95 - 100	Sand or clay	'Tora'	14 - 53	Dimitriou and Aronsson, 2011
Primary	100:9: -	4 000	3 900	2 100	188	94 / -	43 / -	47 / -	Silt	<i>S. spp.</i>	-	Perttu and Kowalik, 1997
Primary	100:67:54	447	558	83	56	- / 28	- / 2	- / 86	Silty and clay soils	'Jorr'	7	Larsson et al., 2003
	100:66:54	893	1 032	167	111	- / 42	- / 55	94			8	
	100:67:54	1 339	1 506	250	167	- / 57	- / 69	96			9	
Primary	100:14: -	160	-	29	4	- / -	- / 58	- / 70	Sandy loam	<i>S. viminalis</i> , <i>S. dasyclados</i> , 'Gudrun', 'Tora'	5	Holm and Heinsoo, 2013
Primary	100:10:13	1 160 (L1)	1 440	366	37	97 / 98	93 / 94	98 / 98	Loamy sand	<i>S. miyabeana</i> 'SX67'	22	Lachapelle-T. et al., 2019
	100:10:13	1 820 (L2)	2 255	579	58	97 / 98	85 / 87	98 / 98			26	
Secondary + coarse filtration + fertilization	100:25: -	300	-	127	32	- / -	97 / -	87 / -	Sandy loam	<i>S. miyabeana</i> 'SX67'	-	Guidi Nissim et al., 2015
	100:20: -	467	-	164	33	- / -	86 / -	83 / -			-	
	100:15: -	735	-	231	34	- / -	95 / -	86 / -			-	

Table 6. Comparison of removal efficiency values to those of the literature data

Nitrogen

As for organic matter removal, the high TKN load removal efficiency achieved for L1 and L2 ($98 \pm 1\%$ and $96 \pm 6\%$, respectively) denotes a high removal capacity of SRWC vegetation filter systems operated in coarse soil which favored aerobic bacterial activity, notably nitrification (Havlin et al., 2013). Water content conditions favorable to nitrification were also maintained during the experimental period. The average WSPS of $65 \pm 17\%$ measured at the L0-2 plot during the first thirteen weeks of the experiment falls within the upper range of 10 to 80 % required by soil nitrifying bacteria and is near the optimum of 60 % for this process (Paul, 2007). The increase in WSPS in the last three weeks of the experiment at an average level of $88 \pm 7\%$ did not result in a decrease in TKN removal efficiency. Other processes of NH_4 transformation or immobilization are unlikely to have played a significant role in TKN removal. The adsorption of ammonia on soil particles was probably negligible due to the low soil clay content at the experimental site (4 %). Soil analysis results also showed that there was no significant accumulation of NH_4 in the soil following the application of L1 and L2 loadings. The volatilization of a significant amount of NH_3 was also unlikely due to the soil pH that was well below the threshold of 7.5 from which this process becomes significant (Havlin et al., 2013).

The difference between total nitrogen removal efficiencies achieved for L1 and L2 suggests that an increase in nitrogen loading led to a decrease of TN removal efficiency and thus, that nitrogen was the limiting design parameter of SRWC operated on coarse soil.

Denitrification and plant uptake are the two main processes of soil nitrate transformation. The irrigation rate applied during the experiment (10 mm/d) and coarse nature of the soil site (loamy sand) did not allow the establishment of soil moisture conditions optimal for denitrification, i.e. a WFPS greater than 80% (Paul, 2007). However, WFPS reached the minimum denitrification threshold of 60% for an average duration of one hour per day, which could have allowed some soil

denitrification. Some denitrification in soil microsites may have taken place under anoxic conditions in the presence of nitrates and biodegradable organic matter (Sylvia et al., 2005). The increase of WFPS to an average value of 88 ± 7 % during the last three weeks of the experiment seems to have been beneficial to denitrification and TN removal efficiency as a drastic decrease in soil pore water nitrates concentration was observed during this period (Figure 3).

The primary effluent properties also appear to have been beneficial to denitrification. The average BOD₅ to TKN ratio of 4:1 measured at the influent of the SRWC during the experiment was greater than the 3:1 ratio typically considered sufficient to support denitrification within the soil of a SR land application system (Reed et al., 1995). This suggests that the carbon input induced by the primary effluent irrigation was sufficient to support soil denitrifying activity and thus, that carbon was not the limiting parameter for denitrification. The absence of a significant difference between soil OM and TOC content at the end of the experiment for L0, L1 and L2 plots concurred with this explanation as this indicates that denitrifying bacteria did not have to draw on soil organic matter content to meet their requirement (Table 5).

The absence of predominant optimal anoxic conditions during the experiment suggests that willow uptake contributed significantly to nitrate removal. The L1 and L2 soil pore water TN concentration increases at the start of September, which corresponds to the willows transpiration decline period (Figure 3), indicates a decrease in nitrate uptake by willows at this period of the experiment. This is consistent with the known link between plant nutrient uptake pattern and transpiration seasonal trend (US EPA, 2006). Early September also corresponds to the end of the willows active growth period in Québec, which typically takes place from May to August (Labrecque and Teodorescu, 2003).

The TN removal efficiency achieved for the L1 loading are close to those reported by Dimitriou and Aronsson (2011) and Guidi Nissim et al. (2015) following the application of similar nitrogen

loadings on willow SRCs (Table 6). Low biomass yields ($< 10 \text{ t DM ha}^{-1} \text{ yr}^{-1}$) and the resulting low nitrate plant uptake may explain the lower efficiency reported by Larsson et al. (2003) (2 % to 69 %) and by Holm and Heinsoo (2013) (58 %). The combined effect of high soil nitrogen background content and lower TN loading rate could also partly explain the lower efficiencies reported by these authors. In addition, the low HLR applied by Holm and Heinsoo (2013) (160 mm/yr) may also have adversely affected the establishment of anoxic conditions favorable to denitrification. The TN loading of $2\,100 \text{ kg N ha}^{-1} \text{ yr}^{-1}$ applied by Perttu and Kowalik (1997) is likely to have exceeded the maximal efficiency of willow SRC regarding TN removal, which may explain the efficiency of 43% reported by these authors.

As for BOD₅ and COD, there are currently no TN or nitrate standards in Québec for municipal wastewater treatment systems that are discharged by soil infiltration. The results obtained were therefore compared to the Québec drinking water quality standards of 10 mg N/L for nitrites and nitrates (MDDELCC, 2015a). The average soil pore water TN concentrations measured through the experiment (2.0 ± 2.7 and $4.5 \pm 6.0 \text{ mg N/L}$ for L1 and L2) were below this standard. NOx concentrations above 10 mg N/L, however, were measured in L1 (two occurrences of 10.3 and 12.3 mg N/L) and L2 (nine occurrences ranging from 10.8 to 22.5 mg N/L) soil pore water from early-September to mid-October. The use of SWRC vegetation filter for the treatment of wastewater is a solution for small rural communities for which the supply of drinking water, in Québec, is often from underground sources (MDDELCC, 2016). SWRC should therefore be designed and operated to minimize the percolation of water with NOx concentration greater than the maximum concentration prescribed by the drinking water quality standards.

Phosphorus

Soil adsorption and precipitation, and plant uptake are the main processes of phosphorus transformation and immobilization in soil. Unlike for TN, no increase in TP or o-PO₄ soil pore

water concentrations occurred in early September, which suggests that soil adsorption and precipitation compensated for the presumed decrease in P plant uptake by willows at the end of their active growth period.

The moderately acidic nature of the soil at the experimental site (pH of 5.6 to 5.8) indicates that soil P immobilization was mainly due to adsorption at the surface of iron or aluminum oxides and to precipitation with soil iron and aluminum minerals (Beaudin et al., 2008; Havlin et al., 2013). The significant decrease in soil exchangeable iron following the L2 treatment suggests a decrease in soil ability to capture phosphorus which could lead to an eventual phosphorus soil profile saturation should wastewater irrigation be continued over a number of years. Such saturation would reduce the SRWC phosphorus removal efficiency and could require some chemical phosphorus removal process upstream of the SRWC.

The almost complete removal of TP (98%) achieved for both L1 and L2 are consistent with the near-complete phosphorus removal expected from land application system prior to the soil profile saturation in P (US EPA, 2006). These removal efficiencies are also close to those reported by Dimitriou and Aronsson (2011) following the application of a similar phosphorus loading on a willow lysimeter (Table 6). Almost complete P removal was also reported by Larsson et al. (2003) following the application of up to $167 \text{ kg P ha}^{-1} \text{ yr}^{-1}$. These results were achieved despite low biomass yields of $7 \text{ to } 9 \text{ t DM ha}^{-1} \text{ yr}^{-1}$, which suggests a P removal mainly attributable to soil adsorption and precipitation, which highlights the importance of the soil texture on short-term phosphorus removal. Similarly, the low removal efficiency reported by Holm and Heinsoo (2013) could have been due to the coarse nature of the irrigated soil, whose low adsorption and phosphorus precipitation potential could not compensate for the low biomass yields achieved ($5 \text{ t DM ha}^{-1} \text{ yr}^{-1}$). As for nitrogen, the TP loading applied by Perttu and Kowalik (1997; $188 \text{ kg P ha}^{-1} \text{ yr}^{-1}$) is

likely to have exceeded the maximal retention capacity of a silty soil SRWC regarding TP removal, which may explain the relatively low efficiency of 47% published by these authors.

There are currently no phosphorus standards in Québec for municipal wastewater treatment systems that are discharged by soil infiltration. The results obtained were therefore compared to the municipal river water discharge standard of 1 mg P/L for TP (MDDELCC, 2015c). The average TP concentrations of soil pore water obtained for L1 and L2 (0.07 ± 0.02 and 0.08 ± 0.03 mg P/L) are well below this standard.

5. Conclusions

The objectives of this study, which was carried out at pilot scale in the summer of 2016 on a two-hectare willow experimental plantation located in Québec, Canada, were to evaluate the treatment efficiency of an SRWC for the treatment of wastewater from a municipal primary effluent in a humid continental climatic context and to establish the design and operation criteria for this process.

Conclusions from this research are:

- Irrigation of a plantation at a constant daily hydraulic loading rate caused an imbalance between irrigation and willows needs. Modulation of irrigation on a seasonal basis according to transpiration trends could allow a better allocation of water and nutrients according to plant needs, and in doing so, result in an increase in total evapotranspiration and nutrient absorption by an SRWC vegetation filter.
- High organic matter removal efficiencies were observed (91 % of COD for L1 and L2). COD concentrations measured in the lysimeters of L1 and L2 plots were similar to the influent S_U , suggesting that an almost complete oxidation of biodegradable organic matter

took place and that the organic loading was not the limiting design parameter of an SRWC vegetation filter operated on sandy soil.

- High ammonia removal efficiencies were observed (98 % of TKN for L1 and L2), suggesting that ammonia removal was not the limiting design parameter of an SRWC vegetation filter operated on sandy soil.
- TN removal efficiencies of 94 and 87 % were observed for L1 and L2 loadings, respectively. Several exceedances of Québec drinking water quality standards for nitrites plus nitrates (10 mg N/L) were observed at the end of the experiment due to the high L2 loading irrigation. These observations suggest that TN loading was the limiting design of an SRWC vegetation filter operated on sandy soil.
- TP removal efficiencies of 98% were observed for L1 and L2 loadings. These results suggest that TP loading was not the limiting design parameter of SRWC vegetation filters. However, a significant increase in soil phosphorus content was observed following L2 loading irrigation (extractable P: L0 = 87 ± 9 and L2 = 96 ± 8 mg P/kg soil). Eventually, after some years of wastewater irrigation, phosphorus soil saturation could limit the removal efficiency of this nutrient, thereby requiring chemical phosphorus removal upstream of the plantation.

This research highlighted that the use of SRWC vegetation filters for the treatment of municipal primary wastewater is a promising solution that could enable small and isolated temperate climate communities to effectively treat and valorize their wastewater. These communities will have to install retention ponds, if they are not already in place, to ensure the primary treatment of wastewater as well as to store wastewater during winter months, in periods of ground freezing and

plant dormancy. SRWC vegetation filters will therefore have to be designed to allow the irrigation of wastewater stored during the winter as well as that generated during the irrigation season.

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